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IS 4006-2 (1985): Methods of test for paper and pulp based packaging materials : Part II [CHD 15: Paper and its products]



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Indian Standard
METHODS OF TEST FOR
PAPER AND PULP BASED PACKAGING
MATERIALS, PART 2
(*First Revision*)

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard
METHODS OF TEST FOR
PAPER AND PULP BASED PACKAGING
MATERIALS, PART 2
(*First Revision*)

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Indian Standard

METHODS OF TEST FOR
PAPER AND PULP BASED PACKAGING
MATERIALS, PART 2

(*First Revision*)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 30 September 1985, after the draft finalized by the Paper and Pulp Based Packaging Materials Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard is supplement to IS : 4006 (Part 1)-1985* which covered methods for some of the tests which are carried out for paper and pulp based packaging materials to evaluate their quality. Some of the methods prescribed in IS : 1060 (Part 1)-1966†, IS : 1060 (Part 2)-1960‡ and IS : 1060 (Part 3)-1969§ may also be used for these materials. A list of the test methods covered in these standard is given in Appendix A.

0.3 The packaging materials used in the industry are many and varied. They are paper and paper products, textiles, metal and metal foils, plastics and a variety of laminates, wood, glass and ceramics, cushioning materials, strapping and hooping materials, nails, etc. Among these paper and paper products are of major importance.

0.4 In this revised standard, the procedure for measuring the flat crushing resistance has been deleted since this method applies only to corrugated boards and would be incorporated in IS : 7063 (Part 2)-1976||. Spectrophotometric method for the determination of reducible sulphur has been incorporated to serve as a referee method.

*Methods of test for paper and pulp based packaging materials, Part 1 (*first revision*).

†Methods of sampling and test for paper and allied products, Part 2 (*revised*).

‡Methods of sampling and test for paper and allied products, Part 3.

§Methods of sampling and test for paper and allied products, Part 4.

||Methods of test for corrugated fibreboard: Part 2 Edgewise crush resistance of board.

0.5 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes methods for the following tests for paper and pulp based packaging materials:

- a) Odour,
- b) Ply separation,
- c) Puncture, and
- d) Reducible sulphur.

1.2 Should any inconsistency exist between the requirements of this standard and those of the standard for an individual material, the latter shall prevail.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in IS : 4261-1967† shall apply.

3. SAMPLING

3.1 Representative samples for test shall be drawn as prescribed in 3 of IS : 1060 (Part 1)-1966.‡

4. CONDITIONING OF TEST SPECIMENS

4.1 Unless specified otherwise in the individual test methods, all the test specimens shall be conditioned according to 5 of IS : 1060 (Part 1)-1966‡.

5. QUALITY OF REAGENTS

5.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1977§) freshly boiled and cooled, shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Rules for rounding off numerical values (*revised*).

†Glossary of terms relating to paper and pulp based packaging materials.

‡Methods of sampling and test for paper and allied products, Part 1 (*revised*).

§Specification for water for general laboratory use (*second revision*).

6. ODOUR

6.0 General — This procedure specifies the methods that may be employed in assessing the risk of odour from packaging materials causing complaint with packaged foods, drugs, etc. The combined sensations of smell and taste are generally described by the term 'flavour' and whenever a food possesses an 'off flavour' due to acquiring adventitious substances it may be described as 'tainted'. The detection methods prescribed here are concerned with odour and flavour derived from volatile constituents contained in packaging materials. Taint can also be caused by other means, such as the extraction of non-volatile constituents from packaging materials by direct contact with the food but these procedures are not concerned with any form of taint other than that produced by odorous volatile constituents in packaging materials. The recognition of odour or taint is subjective process occurring within the brain of an individual. Hence they are not objective qualities and cannot be assessed without the judgement of a human observer.

6.1 Test Panel — It is desirable to organize a test panel, which may consist of four to eight members, selected carefully from those who have demonstrated an acute and discerning sense of smell. The senses of taste and odour are closely allied and members of the panel should also be tested thoroughly for taste sensitivity and differentiation. Taste is a usefull method of detecting odours as described under 6.5.

6.2 Sample Containers and Testing Room

6.2.1 Sample Containers — Vapourproof containers may be necessary for sampling, storage and holding test pieces for the development of maximum intensity of odour. Containers shall be clean, dry and free from odour. Aluminium foil or cellulose film shall be substituted for any rubber gaskets or stoppers. Samples may also be wraped directy in foil, cellulose film or vegetable parchment.

6.2.2 Testing Room — Shall be free from plant or industrial odours. Extremes of temperatures should be avoided, except in special cases and all distracting influences should be minimized.

6.3 Materials

6.3.1 Standard Samples — In some distances, standard samples, representing satisfactory and maximum permissible levels of odour, may be set up. Maintenance of the samples is a serious problem, since age and other factors may alter odour drastically. In all cases, it is desirable to have some type of reference sample available.

6.3.2 Water (Odour Free) — Moisture is commonly used to intensify odours, or develop potential odours. Normally, tap water is satisfactory,

except where residual odour or a chlorine smell is present. Distilled water is not always odourless. Each portion of water shall be smelled and tasted before acceptance.

6.3.3 Fatty Materials — A range of substances containing oil or fat may be used to pick up certain types of odours, with identification and degree of odour determined both by smell and taste. Common materials are mineral oil, milk chocolate, cream, and fresh unsalted butter.

6.4 Test Specimen — The preparation of the test sample for presentation to the panel is very important. No one standard method of doing this is sufficient, since the type of materials under test and the possible odours present, allow a wide range of possibilities. A few of the methods which have been used are given in **6.4.1** to **6.4.3** as a guide; others may be devised as necessary. Each laboratory should develop and record the detailed preparation procedure found satisfactory for specific products.

6.4.1 Direct Testing — Cut or tear representative samples of the packaging materials to a convenient size. It is often desirable to open up fresh surfaces at the instant of smelling (tearing paperboard, scraping wax with a knife, breaking open glue joint, etc.).

6.4.2 Moistening — Water brings out some types of odours by various mechanisms, and especially when the product may be subjected to moisture, try one of the following.

6.4.2.1 Breathing on the sample — The tester exhales on the test piece, then smells it.

6.4.2.2 Dampening — Sprinkle the sample lightly with water, then smell. Time is sometimes required to develop an odour. Normally, the test piece is dampened, at 21 to 27°C and stored at 38°C, in an odourless glass jar for 24 h and then tested.

6.4.2.3 Soaking — Saturate or submerge the test piece in water for a period of time and at a temperature judge a suitable for each case. Test by smelling both the water and the test piece.

6.4.3 Transfer to Oily Product

6.4.3.1 Place the test piece in a covered glass dish adjacent but not in contact with mineral oil for 4 to 24 h at room temperature. Oil soluble materials, such as printing ink, kerosine, etc., can be detected by smelling the oil.

6.4.3.2 Prepare three sandwiches with a part of unsalted butter between two pieces of test piece and overwrap with parchment, cellulose

film or foil. Hold for 1, 2 and 3 days, respectively, at room temperature; refrigerate for 30 min; then taste and smell the butter. Reference samples may be prepared using odour free parchment.

6.4.3.3 Place the folded sample in a watertight tray and add 3 cm depth of coffee cream (18 percent butter fat). Cover with aluminium foil, hold at 7°C for 24 h, then taste the cream. This procedure is used widely for printing ink odours and is very sensitive. As an alternative, strips of material may be placed in a dish or flask and covered with cream. Run a blank test on cream in a glass bottle with a foil cap.

6.4.3.4 Place the test piece in a covered glass dish adjacent to a piece of plain milk chocolate, and hold at room temperature for 1 to 2 days. Taste the chocolate and compare with a control.

6.4.3.5 Prepare a package by wrapping same commercial product, known to be sensitive to odour, with the test piece materials. Hold for the desired time and temperature, then smell and taste the product in comparison with a control.

6.5 Procedure — Samples shall be identified only by code numbers, including the control, and tests should be given only the minimum amount of information prior to the test. Each member shall be given a set of test pieces and a report form, and they shall work independently. Table 1 may serve as a procedural guide.

6.6 Report — Each individual panel member shall report independently. Complete agreement by the panel may be expected but the group report serves as valuable data.

6.6.1 Unknown Odour — Type, its chemical nature and origin (if possible).

6.6.2 Known Odour — Rated as:

- a) essentially none (pleasant);
- b) slight but not objectionable (neutral);
- c) moderate, objectionable (unpleasant); and
- d) strong (offensive).

6.6.3 Taste as Odour Index — The same reporting system shall be used as for smelling.

7. PLY SEPARATION OF COMBINED CONTAINER BOARD

7.1 General — This method evaluates the resistance to ply separation of laminated solid fibre or combined corrugated board fabricated with water resistant adhesives.

TABLE 1 COMMON PACKING MATERIALS, ODOURS AND DETECTION METHODS

(Clause 6.5)

SL No.	MATERIAL	TYPE OF ODOUR	TEST METHOD, REF TO CL No. IN STANDARD
(1)	(2)	(3)	(4)
i)	Synthetic film	Inherent solvent, plasticizer, monomer, etc	6.4.1 (24 h at 38°C), 6.4, 3.1 and 6.4.3.5
ii)	Paper and board	Inherent kraft Musty or mouldy Chlorinated phenol Sour	6.4.1, 6.4.2.1 and 6.4.2.2 6.4.1, 6.4.2.1 and 6.4.2.2 6.4.1 and 6.4.2.3 6.4.1 and 6.4.2
iii)	Paper, coated	Volatile additives, casein decomposition	6.4.1
iv)	Glassine, coated	Inherent in coating	6.4.1 (24 h at 38°C), 6.4.3.1 and 6.4.3.5
v)	Ink and varnish	Oxidizing oils, plasticizer, solvent	6.4.1 and 6.4.3.3
vi)	Adhesive joints (after thorough drying)	Solvents, sour, starch added perfume	6.4.1 (fresh and after 24 h at 38°C)
vii)	Wax	Oil, residual oxidized solvent	6.4.1 (fresh shavings at room temperature in small containers); and 6.4.3.5 (Cheese, for example)

7.2 Apparatus

7.2.1 Water Tank — Of corrosion-resisting metal. Suggested minimum dimensions are 40 cm length, 30 cm breadth and 25 cm height. Rods for holding the test pieces 25 mm under the surface of the water are attached 20 cm from the bottom of the tank and 5 cm from each of the 40 cm sides. Some means for keeping the test pieces separated from each other and from the side walls of the tank should be provided.

7.2.2 The tank shall be thoroughly washed for each test. Soap shall not be used for washing since any residue will seriously affect the test.

7.3 Materials

7.3.1 Water — Potable tap water at a temperature of $27 \pm 2^\circ\text{C}$. It shall have a pH value between 6.5 to 7.5 and shall be drawn fresh for each batch of test pieces. Care shall be exercised to eliminate air in the water.

7.4 Test Specimens

7.4.1 The test specimens shall preferably be 15×25 cm in size. At least four test pieces of each sample shall be tested.

7.4.2 The sample shall be allowed to cure in an atmosphere of 65 ± 2 percent relative humidity at a temperature of $27 \pm 2^\circ\text{C}$ for at least one week from the time of manufacture before test. For control purposes, at the time of manufacture a quick test of board may be made by accelerated curing in an oven for 10 to 20 min at a temperature of 105°C prior to placing the test pieces in water.

7.5 Procedure — Submerge the test pieces virtually within 25 cm edges horizontal and the top edge 25 mm below the surface of the water. Support the test pieces so that the water has free access to all surfaces and ply separation is not restricted. After immersion for 24 ± 0.25 h, remove the test pieces from the tank and soak off excess surface water by suitable means, such as blotters or paper towels. Examine the test pieces immediately for ply separation and satisfactory bonding. Should some separation of the component plies appear at the edge of the board, such separation shall not extend more than 6 mm from the edge when flexed by pressure from the thumb. The plies must not be forced apart. They should be flicked lightly at the edge with the thumb. All other areas shall be satisfactorily bonded.

7.6 Report — The report shall include the following:

- a) Number of test pieces tested;
- b) Number of test pieces which failed; and
- c) Dimensions of the test pieces.

8. PUNCTURE TEST

8.0 General — This method covers the procedure for puncture test of paper board, corrugated and solid fibreboard. The test is a measure of the energy required to force a puncture head of designated size and shape completely through a sample of fibreboard. The measured value is dependent upon the resistance to puncture at the stiffness of the board.

8.1 Apparatus — Beach puncture test apparatus, consisting of the parts given in **8.1.1** to **8.1.9**

8.1.1 Pendulum — Incorporating an arm in the form of an arch of 90° .

8.1.2 Puncture Point — Affixed to the end of the arm and having the shape of a right angled triangular pyramid 25 mm in height, base edges

left sharp with round leading edges and point honed to 1.57 ± 0.05 mm radius.

8.1.3 Puncture Point Collar — Loose fitting around the base of the puncture point to eliminate friction that would result if the punctured material rubbed against the puncture arm.

8.1.4 Trigger or Latch — To hold the pendulum in the horizontal position.

8.1.5 Clamping Plates — Spring actuated, for holding the test piece rigidly at the level of axis of the pendulum.

8.1.6 Pointer and Scale — To indicate the arc through which the pendulum travels after penetrating through the test piece.

8.1.7 Weights — Which when attached, give a maximum scale value of at least 1 300 units of energy when the pendulum is in the horizontal position and scale graduations to correspond to the weights used.

8.1.8 Loose Plate — Inserted between the clamping plates when testing the material for the purpose of defining the test area. This plate shall be removed when checking the zero reading of the apparatus.

8.1.9 Adjustable Screw — Bearing against the pointer assembly during the measuring half of the pendulum swing, which permits adjustment of the full scale position of the pointer.

8.2 Adjustment for Calibration of Apparatus

8.2.1 Place the apparatus on a rigid heavy foundation of convenient height and level it in both the horizontal directions.

8.2.2 Before starting calibration of the apparatus it is important that the entire clamping assembly should be unbolted and moved away from the path of pendulum.

8.2.3 To check the zero reading, remove the weights and the stud that supports the weight. Set the pointer about 25 mm above zero. Place the loose collar above the base of the puncture head. Raise the pendulum to the cocked position and release it. Note the pointer reading. Repeat this operation several times. The pointer should register zero with each attempt. If it is not the case, make suitable adjustment of the zero adjustment screw, located on the arm extending from the bearing housing of the pendulum, until the zero reading is obtained.

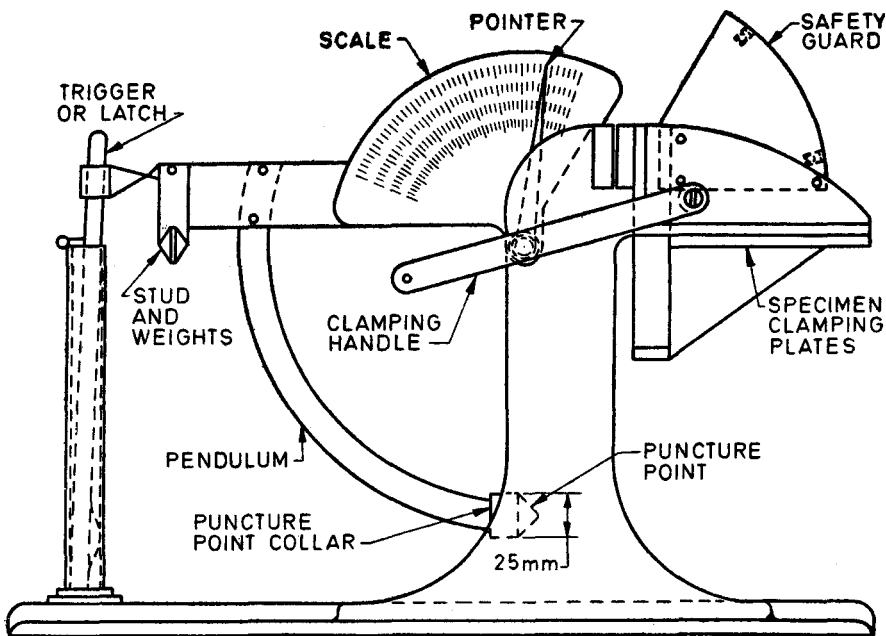


FIG. 1 PUNCTURE TESTER

8.2.4 Check the friction of the pointer by the latching the bare pendulum, without the weights or weight stud, and with the collar attached to the puncture head. Set the pointer at full scale position before releasing the pendulum from the cocked position. After the pendulum has completed its swing, the pointer should not be more than 3.2 mm above the zero position. An error of more than 3.2 mm indicates excessive friction which can be adjusted by relieving the spring pressure between the pointer collar and the shaft. The pressure is governed by the positions of the screw in the stud projecting from the pointer hub.

8.2.5 The puncture point collar is held in place on the base of the puncture head by a pin containing a small ball under spring pressure. The position of this pin in the puncture head base is adjustable and can be locked by means of a set screw. The position of this pin and ball should be such that the loose sleeve is very lightly held on the base of the puncture head, and can be removed with a minimum of energy. One of the three possible positions of collar on the base is chosen and marked on the side for use of collar always in the same position.

8.3 Procedure

8.3.1 Place the conditioned test piece between the clamping plates. Before making each test, place the puncture point collar on the base of the puncture head and set the pointer about 25 mm above the expected reading. Raise the pendulum to the horizontal position. Release by pushing the latch handle to the left. Note the reading on the proper scale after the pendulum has completed its swing. This reading should be made to the nearest one-half division of the scale used. It is not advisable to make tests giving values within 25 percent of the upper or lower end of the scale. In such cases use another suitable scale.

8.3.2 Orient the test pieces in such a manner that the edge of the puncture point, which is in the plane of the puncture arm, is perpendicular to the corrugations of combined corrugated, or grain direction of the uncombined sheets, in half the number of test pieces. Make equal number of tests with either surface of the board facing up. On uncombined board or single face corrugated board, make all tests with smooth surface down.

8.4 Report — The report for puncture test shall include the following:

- a) Room temperature and relative humidity;
- b) Scale used;
- c) Number of tests made each way of material;
- d) Average results for test made each way of the material;
- e) Total average results, accurate to 2 kgf.cm if the average value is more than 100 kgf.cm, accurate to 1 kgf.cm, if the average value is between 50 and 100 and to nearest 0.5 kgf.cm, if the average value is less than 50 kgf.cm; and
- f) Maximum and minimum values obtained.

NOTE — 1 beach unit = 0.305 cm kg.

9. REDUCIBLE SULPHUR

9.0 General — This method describes a quantitative and a semi-quantitative procedure for the determination of reducible sulphur in paper and paper board. The semi-quantitative test indicates the general level of reducible sulphur with limited accuracy while the quantitative procedure yields results of much greater accuracy.

9.0.1 The methods outlined are not necessarily a measure of how much a given test material will tarnish polished metals. When tested by this

method, paper which has less than 0.000 8 percent reducible sulphur may be assumed to be non-tarnishing as far as sulphur is concerned, but if more than 0.000 8 percent, this does not necessarily mean tarnishing will occur, because sulphur compounds which may not cause staining are reduced by the treatment with the subsequent evolution of hydrogen sulphide.

9.0.2 If the *pH* of the paper is low, that is, 4 to 4.5 (cold extraction), as little as 0.000 2 percent of reducible sulphur may cause tarnishing of initiation gold bronze prints; whereas, if the *pH* were higher, for example, on brush-coated art paper, a much higher quantity of sulphur might be tolerated.

9.0.3 The procedures described do not give the total sulphur content, nor the total reducible sulphur content, nor are they intended to. They are limited to the reducible sulphur content determined under the conditions of the test (for instance a fairly mild reduction system). Those forms of sulphur believed to be 'reducible' under the test condition are (a) sulphide (released by the acid, not actually reduced), (b) elemental sulphur, (c) thiosulphate, (d) other polythionates or polysulphides, (e) perhaps sulphide. Sulphate is excluded.

9.0.4 Great care shall be taken to avoid possible contamination of the reagents and test pieces by contact with the bare fingers or minute quantities of sulphur compounds being absorbed from the atmosphere. It is advisable to run a blank test using the same apparatus and reagents to ascertain that they do not contain, nor have acquired any appreciable quantity of sulphur.

9.1 Quantitative Method

9.1.1 Apparatus

9.1.1.1 Reaction apparatus — Consisting of a 500-ml round bottomed three necked flask, equipped with a gas inlet tube adapter with perforated bulb, a 60-ml pear-shaped funnel (dropping funnel), a 200-mm condenser with a side-arm adapter. A heating mantle with variable power supply forms part of the reaction apparatus.

9.1.1.2 Absorption apparatus — Consisting of a 100 ml amber or low actinic flask in a suitable ice bath.

9.1.1.3 Colorimeter or spectrophotometer — To read at 660 to 670 nm equipped with 20 to 50 mm cuvettes.

9.1.1.4 Pipettes — 1, 2, 5, 10 ml serological or bacteriological (blow out).

9.1.1.5 *Disintegrator* — A blender or other such rotating blade device.

9.1.1.6 *Water bath* — At 100°C or steam bath.

9.1.2 Reagents

9.1.2.1 *Aluminium foil* — Not over 0.25 mm thick, sulphur free.

9.1.2.2 *Phosphoric acid* — 85 percent, sulphur free.

9.1.2.3 *Cadmium hydroxide absorption suspension* — Dissolve 4.3 g of cadmium sulphate ($3 \text{ Cd. SO}_4 \cdot 8 \text{ H}_2 \text{ O}$) in water; add 0.3 g of sodium hydroxide, dissolved in water and 10 g of arabino galactan; dilute to 1 000 ml and stir well. This is a saturated solution of cadmium hydroxide; the suspension is swirled before pouring off an aliquot.

9.1.2.4 *Ferric chloride solution* — Dissolve 100 g of ferric chloride hexahydrate ($\text{FeCl}_3 \cdot 6 \cdot \text{H}_2\text{O}$) in water and dilute to 100 ml.

9.1.2.5 *Amine Solution:*

a) *Stock amine solution* — Dissolve 25 g of P-amino-dimethylaniline sulphate (that is N, N-dimethyl-P-phenylene diamine sulphate) in 75 ml of cold (1 : 1) sulphuric acid and dilute to 100 ml with the (1 : 1) acid.

b) *Dilute amine solution* — Pipette 2.5 ml of the stock solution into a 100-ml volumetric flask and dilute to the mark with (1 : 1) sulphuric acid.

9.1.2.6 *Sodium sulphide standard solution:*

a) *Stock solution* — Dissolve 0.4 g sodium sulphide hexahydrate ($\text{Na}_2 \text{ S} \cdot 9 \text{ H}_2\text{O}$) (use large crystals which appear dry) in water deaerated with nitrogen. Dilute to 250 ml with the deaerated water. After the solution has been diluted to the volume, add 5 to 10 ml of cyclohexane to protect the solution from air oxidation. The solution is likely to be stable for one month.

b) *Working solution* — (to be prepared daily). Pipette 10.0 ml of stock solution into a 1 000 ml volumetric flask and dilute to the mark with the deaerated water. One ml of this solution contains 2 µg of sulphur.

NOTE — This solution or the stock solution may be standardized by potentiometric titration with silver nitrate solution using a sulphide specific electrode; the sulphide standard should be at least 90 percent pure.

9.1.2.7 *Nitrogen gas, sulphur free*

9.1.3 *Test Specimen* — A minimum of 6 g of sample is required to provide triplicate test specimens. From each test unit of the sample, cut

and weigh 2 g to the nearest milligram for each test specimen. Determine the average moisture content of the paper.

9.1.4 Procedure

9.1.4.1 Disintegrate the sample in the blender with 75 ml of water and transfer the test specimen from the disintegrator quantitatively to the 500 ml reaction flask using additional aliquots of 5 ml of water to assist in the transfer. Add 2 g of aluminium foil which has been cut into 13 mm square and complete the assembly of the reaction apparatus.

9.1.4.2 *Precipitation of cadmium sulphide* — Measure 10 ml of the phosphoric acid into the dropping funnel and 75 ml of the cadmium hydroxide absorption suspension (which is swirled before pouring and should include some solids) into the darkened 100-ml volumetric collecting flask. Start bubbling the nitrogen at the rate of about 5 bubbles per second, taking care to ensure that there are no leaks. Use top quality sulphur-free stop cock grease at the joints and if necessary tie the joints with clamp or rubber bands. After the nitrogen has been adjusted, submerge the delivery tube into the absorption suspension in the 100-ml volumetric flask and put the ice bath in place, taking care to ensure that the tip is to the bottom of the flask, that is, submerged as deeply as possible. Add the 10 ml of phosphoric acid from the dropping funnel. Leave a small amount of acid in the tip. Close the stop cock on the funnel, and start heating the mixture to a boil. Take care that the absorption solution does not back up in the delivery tube while the mixture is getting heated. It may be necessary to adjust the nitrogen flow to prevent this. Discontinue heating after 45 min, remove the volumetric flask together with the side-arm adapter by dislodging it at the condenser, and stop the nitrogen flow. Keep the adaptor together with the flask until the amine reagent is added in the colour development step because the cadmium sulphide often hangs up in the tube tip. The sample should not be stored in this manner for more than one hour.

9.1.4.3 *Preparation of calibration standard* — (To be prepared each time the analysis is performed.) Add 75 ml of the cadmium hydroxide absorption suspension to each of five 100 ml darkened volumetric flasks and place in the constant temperature bath (20 to 25°C). Pipette 1, 2, 5 and 10 ml respectively of the sodium sulphide standard working solution into four of the volumetric flasks. The standards contain 2, 4, 10 and 20 µg of sulphide per 100 ml. The flask containing no sulphide is the reagent blank.

NOTE — The blow-out pipettes are used to pipette the sodium sulphide working solutions aliquots. They are immersed into the absorption solutions and allowed to drain slowly while the tips are below the surface. When drainage is complete, gently blow out the last drops while still immersed and then remove from the flasks.

9.1.4.4 Colour development and read out — To the test solution reagent blank and calibration, standards in 100-ml volumetric flasks, add 2 ml of the diamine solution. Allow the dense amine solution to flow down the side of the flask to the bottom of the absorption solution. Stopper the flask and swirl gently but adequately a few minutes without shaking. Immediately add 10 drops of the ferric chloride solution and shake vigorously to react any hydrogen sulphide which may have come out of solution. Dilute each solution to the mark with water, mix well, and allow to stand for atleast 20 minutes. The colour development should be conducted in the dark and if possible at 20 to 25°C by means of the constant temperature bath. Read the maximum absorbance at the peak or near 670 nm, using water as the reference. Subtract the absorbance of the reaction blank (as the correction) from the calibration standards and the test specimens. Plot the corrected absorbance of the calibration standards against µg of sulphur present and draw the best straight line passing through zero. (The slope should be about 0.005 to 0.010 absorbance units per µg of sulphur per 10 nm of path length.) Read the amount of reducible sulphur, in micrograms, in each specimen directly from the chart based on its net absorbance.

9.1.5 Calculation

9.1.5.1 Percentage reducible sulphur

$$= \frac{(\mu\text{g reducible sulphur from chart}) \times 0.0001}{\text{Mass of specimen in grams}}$$

9.2 Semi-Quantitative Method

9.2.1 Apparatus

9.2.1.1 Reaction apparatus — Consisting of a 100-ml flask carrying a short, water-cooled condenser as shown in Fig. 2. A flanged head containing a disc of filter paper impregnated with lead acetate is placed over the condenser. The condenser prevents water vapour from condensing on the test paper which would cause an uneven development of the spots.

9.2.1.2 Pipettes — 1-ml graduated in 0.01 ml divisions and 10 ml graduated in 0.1 ml.

9.2.1.3 Water bath — At 100°C, or steam bath.

9.2.2 Reagents

9.2.2.1 Aluminium foil — not over 0.25 mm thick, sulphur free.

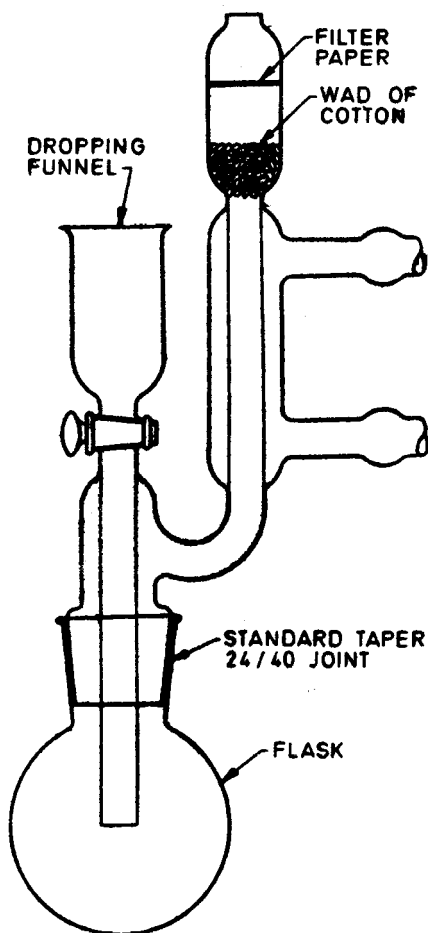


FIG. 2 REACTION APPARATUS

9.2.2.2 Standard sodium thiosulphate solution — Dissolve 0.097 0 g of fresh, clean crystals of sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) in a little water in a 100 ml volumetric flask, dilute to mark and mix thoroughly. Dilute 10 ml of this solution to 1 litre and mix thoroughly. One ml is equivalent to 0.002 5 mg of sulphur.

9.2.2.3 *Lead acetate solution* — 20 percent.

9.2.2.4 *Test paper* — Rapid filtering grade of filter paper untouched with bare fingers and freshly impregnated with the lead acetate solution, dried and cut into 25 mm diameter discs stored in an air-tight container.

9.2.2.5 *Phosphoric acid* — 85 percent, sulphur free.

9.2.3 *Test Specimen* — Cut pieces of paper about 6 mm square from representative sample sheets and weigh the equivalent of 0.25 ± 0.005 g of moisture free paper. Determine the average moisture content of the paper if it is not known to be within 1 percent. For papers which are low in sulphur, increase the weight of specimen preferably to 0.5, 0.75 or to a maximum of one gram. The sample for test and the test pieces shall be handled only with clean forceps.

9.2.4 *Procedure* — Transfer the weighed test piece to the flask by means of forceps. Add a few millilitres of water and macerate the paper by a clean glass stirring rod until it is thoroughly soaked and at least partially disintegrated. Wash down the glass rod with a few millilitres of water until the total volume in the flask is about 20 ml. Add 2 g of aluminium foil cut into small pieces. Fit the funnel and the condenser to the flask. Insert in the neck of the condenser a loose wad of surgical cotton. Attach the head containing the disc of lead acetate paper, moistened with a minimum amount of water over the condenser. Through the dropping funnel add 10 ml of the phosphoric acid and close the cock. Provide arrangements for the introduction of stream of inert gas in the flask to remove the evolved hydrogen sulphide gas. Heat the flask on the water bath for 45 min. Remove the apparatus and take out the lead acetate disc. Compare it with the set of discs stained with known amounts of sulphur prepared as described below and estimate the amount of sulphur evolved by the test piece. The set of stained discs shall be prepared using the same chemicals and procedure except that in place of the test piece measured amounts of the standard sodium thiosulphate solution, such as 0.1, 0.2, 0.4, 0.6, 0.8 and 1.0 ml respectively are added. This will give a range suitable for most papers. One millilitre of the solution contains the equivalent of 0.001 percent sulphur in a 0.25 g test piece.

9.2.5 *Report* — The amount of reducible sulphur shall be reported as a percentage by mass of the air-dry paper.

APPENDIX A(*Clause 0.2*)**METHODS OF TEST FOR PAPER AND PULP BASED
PACKAGING MATERIALS**

<i>Method of Test for</i>	<i>Ref to Cl. No.</i>
Abrasion loss	9 of IS : 4006 (Part 1) - 1985
Air permeance	13 of IS : 4006 (Part 1) - 1985
Alkali staining number	11 of IS : 1060 (Part 3) - 1969
Alkalinity	12 of IS : 1060 (Part 3) - 1969
Alpha cellulose content	15 of IS : 1060 (Part 2) - 1960
Ash	11 of IS : 1060 (Part 1) - 1966
Bending test	8 of IS : 1060 (Part 2) - 1960
Benzene of soluble matter, estimation of	21 of IS : 1060 (Part 2) - 1960
Bleeding resistance	11 of IS : 1060 (Part 2) - 1960
Blocking resistance	10 of IS : 4006 (Part 1) - 1985
Breaking length	12.4 of IS : 1060 (Part 1) - 1966
Brightness	13 of IS : 1060 (Part 2) - 1960
Bulk	8 of IS : 1060 (Part 1) - 1966
Bursting strength	12.5 of IS : 1060 (Part 1) - 1966
Bursting strength, wet	6 of IS : 1060 (Part 2) - 1960
Chlorides, water soluble, estimation of	17 of IS : 1060 (Part 2) - 1960
Cobb test	13.2.2 of IS : 1060 (Part 1) - 1966
Compression resistance (ring crush)	11 of IS : 4006 (Part 1) - 1985
Copper number	13 of IS : 1060 (Part 3) 1969
Cupping test	9 of IS : 1060 (Part 2) 1960
Ether soluble matter, estimation of	20 of IS : 1060 (Part 2) - 1960

IS : 4006 (Part 2) - 1985

<i>Method of Test for</i>	<i>Ref to Cl. No.</i>
Exudation test	10 of IS : 1060 (Part 2) - 1960
Fastness to light	12 of IS : 1060 (Part 2) - 1960
Fatty and/or rosin acid extractable by ether estimation of	19 of IS : 1060 (Part 2) - 1960
Fibre composition	18 of IS : 1060 (Part 1) - 1966
Flexural resistance and deflection	5 of IS : 4006 (Part 1) - 1985
Folding endurance	12.6 of IS : 1060 (Part 1) - 1966
Gloss	15 of IS : 1060 (Part 1) - 1966
Grease resistance	8 of IS : 4006 (Part 1) - 1985
Lead and lead compounds, estimation of	16 of IS : 1060 (Part 1) - 1960
Moisture content	9 of IS : 1060 (Part 1) - 1966
Oil absorption	17 of IS : 1060 (Part 1) - 1966
Opacity	16 of IS : 1060 (Part 1) - 1966
pH value	10 of IS : 1060 (Part 1) - 1966
Rigidity, stiffness and softness	12 of IS : 4006 (Part 1) - 1966
Stiffness	7 of IS : 1060 (Part 3) - 1969
Substance	6 of IS : 1060 (Part 1) - 1966
Sulphates, water soluble, estimation of	18 of IS : 1060 (Part 2) - 1960
Surface pH	9 of IS : 1060 (Part 3) - 1969
Tearing resistance	12.7 of IS : 1060 (Part 1) - 1966
Tensile strength and stretch	12.3 of IS : 1060 (Part 1) - 1966
Tensile strength, wet	7 of IS : 1060 (Part 2) - 1960
Thickness	7 of IS : 1060 (Part 1) - 1966
Water absorbency	14 of IS : 1060 (Part 1) - 1966
Water penetration	10 of IS : 1060 (Part 3) - 1969 and 7 of IS : 4006 (Part 1) - 1985

IS : 4006 (Part 2) - 1985

Method of Test for

Ref to Cl. No.

Water proofness

6 of IS : 4006 (Part 1) - 1985

Water vapour permeability

14 of IS : 1060 (Part 2) - 1960

Wax absorptiveness

6 of IS : 1060 (Part 3) - 1969

Wax pick number

8 of IS : 1060 (Part 3) - 1969

IS : 4006 (Part 2) - 1985

(Continued from page 2)

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